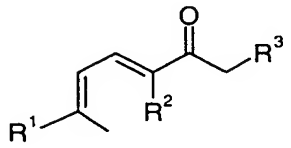
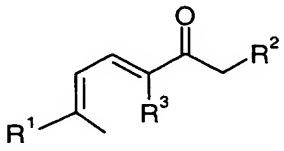
What is claimed is:

1. A continuous process for preparing pseudoionones of the general formulae I or I′ and isomers thereof



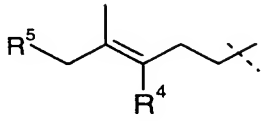
(I)

or



(I′)

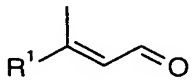
where R^1 is

CH_3 or 

R^2, R^3 are each hydrogen, CH_3 or C_2H_5,

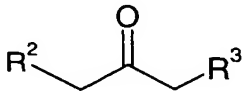
R^4, R^5 are each hydrogen or CH_3,

by reacting an aldehyde of the formula (II)



(II)

with an excess of a ketone of the general formula (III)



(III)

where R^1, R^2 and R^3 are each as defined above, in the presence of water and alkali metal hydroxide at elevated temperature in homogeneous solution, which comprises

a) mixing the homogeneous solution of aldehyde, ketone and aqueous alkali metal hydroxide at a temperature of from 10 to 120°C, then

b) removing the water and alkali metal hydroxide which have not dissolved in the reaction mixture,

c) subsequently passing the homogeneous reaction mixture, avoiding backmixing, at a temperature which is from 10 to 120°C above the boiling point of the lowest-boiling component and a vapor pressure p of from 10^6 to 10^7 Pa through a reactor which enables a residence time of from 2 to 300 minutes,

d) cooling the reaction mixture under decompression,

e) removing the ketone from the reaction mixture with steam in countercurrent and

f) drying the crude product and freeing it of excess aldehyde and secondary components using a rectification column.

2. The process according to claim 1, wherein the ketone component of the general formula (II) is added in a from 5- to 50-fold molar excess, and the unconverted proportion, downstream of the reaction zone, is removed at a pressure of from 10^7 to $5 \cdot 10^8$ $mPa_{abs.}$ and added again to the fresh ketone for the synthesis.

3. The process according to claim 1 or 2, wherein the reaction temperature at a given residence time is selected in such a way that the conversion of the aldehyde component is from 60 to 98%, and the unconverted aldehyde is removed and recycled into the reaction.

4. The process according to claims 1 to 3, wherein the water content of the ketone, used for the reaction, of the formula (III) is between 1 and 15% by weight.

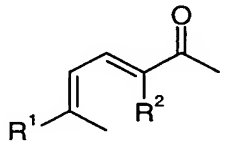
5. The process according to claims 1 to 4, wherein the concentration of the alkali metal hydroxide used for the reaction is between 0.005 and 50% by weight, preferably 5 – 10% by weight.

6. The process according to claims 1 to 5 for preparing pseudoionones of the formula I and isomers thereof where R^2 or R^3 is methyl, wherein the concentration of the alkali metal

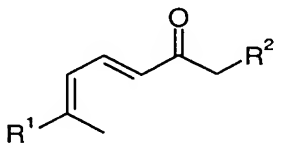
hydroxide used for the reaction is from 10 to 50% by weight, preferably from 35 to 45% by weight.

7. The process according to claims 1 to 6, wherein the ketone of the formula (III) used consists substantially of excess ketone of the formula (III) which has been removed from the reaction and has a water content of 1 – 15% by weight, which may be supplemented with either anhydrous or aqueous ketone of the formula (III) having a water content of 1 – 15% by weight.

8. The process according to claims 1 to 7, wherein, in the case of reaction with ketones of the general formula (III) where $R^2 \neq H$ and $R^3 = H$, a product mixture is obtained which contains from 70 to 95% n-alkylpseudoionones and from 5 to 30% isoalkylpseudoionones

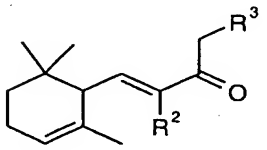


iso - alkylpseudoionone

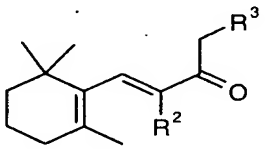


n - alkylpseudoionone

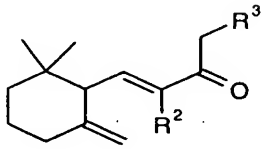
9. A continuous process for preparing ionones of the general formulae (IV), (V) and (VI) and isomers thereof, which comprises reacting the pseudoionones obtained according to claims 1 to 8 to give ionones of the general formulae (IV) to (VI)



α-Isomer
(IV)



β-Isomer
(V)



γ-Isomer
(VI)

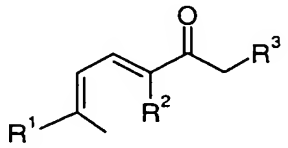
in the form such that the ratio of the n-form ($R^2 = H$, R^3 = alkyl) to the iso-form (R^2 = alkyl, $R^3 = H$) according to claim 8 is maintained.

10. The process according to claim 9, wherein the pseudoionones obtained according to claims 1 to 8 are reacted with highly concentrated sulfuric acid in the presence of a diluent which is inert under the reaction conditions to give ionones, the reaction temperature being 0-20°C and the residence time between cyclization and hydrolysis being from 10 to 300 seconds, preferably 120 seconds.

Continuous preparation of pseudoionones and ionones

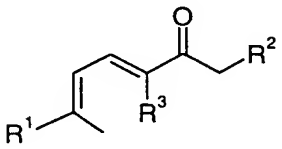
Abstract

A continuous process for preparing pseudoionones of the general formulae I or I′ and isomers thereof



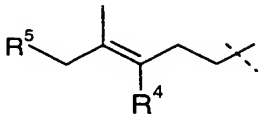
(I)

or



(I′)

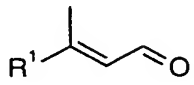
where R^1 is

CH_3 or 

R^2, R^3 are each hydrogen, CH_3 or C_2H_5,

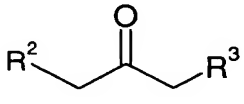
R^4, R^5 are each hydrogen or CH_3,

by reacting an aldehyde of the formula (II)



(II)

with an excess of a ketone of the general formula (III)



(III)

where R^1, R^2 and R^3 are each as defined above, in the presence of water and alkali metal hydroxide at elevated temperature in homogeneous solution, which comprises

a) mixing the homogeneous solution of aldehyde, ketone and aqueous alkali metal hydroxide at a temperature of from 10 to 120°C, then

b) removing the water and alkali metal hydroxide which have not dissolved in the reaction mixture,

c) subsequently passing the homogeneous reaction mixture, avoiding backmixing, at a temperature which is from 10 to 120°C above the boiling point of the lowest-boiling component and a vapor pressure p of from 10^6 to 10^7 Pa through a reactor which enables a residence time of from 2 to 300 minutes,

d) cooling the reaction mixture under decompression,

e) removing the ketone from the reaction mixture with steam in countercurrent and

f) drying the crude product and freeing it of excess aldehyde and secondary components using a rectification column.